Operating procedure for JEOL 7600F High Resolution Analytical SEM

I. Specimen preparation

There are several holders for different kinds of specimens and applications. During your initial training you should have received a general overview of these holders. Also, you should have received training on specimen mounting using the holder that best suits your specific application. **Only use a holder for which you have received training by the tool instructor.** If you wish to use a different holder, first contact the tool instructor.

It is very important to know the kind of holder you are using and the way to mount specimens. For example, for the 12.5 and 26 mm holders, the correct way to mount your specimen is to flush its surface with the cylinder top face (see Fig. 1).

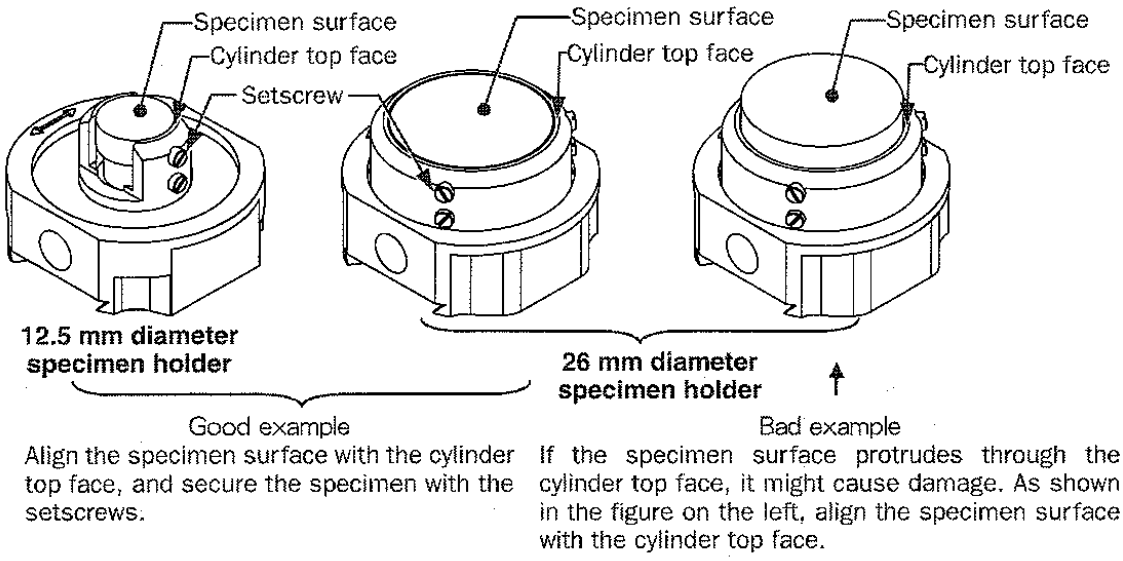

FIG. 1. Specimen positioning on 12.5 and 26 mm holders. (Diagram taken from JEOL's manual.)

If your specimen needs to protrude above the cylinder's top face (or the top face of another holder), you can still use this holder, but you need to estimate (with approx. 1 mm accuracy) the *offset* between the specimen and holder top surfaces. To make sure you are doing things correctly, use the *sample height tool* (see Fig. 2). Try to have the sample's surface aligned with the *zero offset line*. If it needs to be above this line, read the offset in the meter scale. This offset value will be used when loading your sample in the SEM chamber.

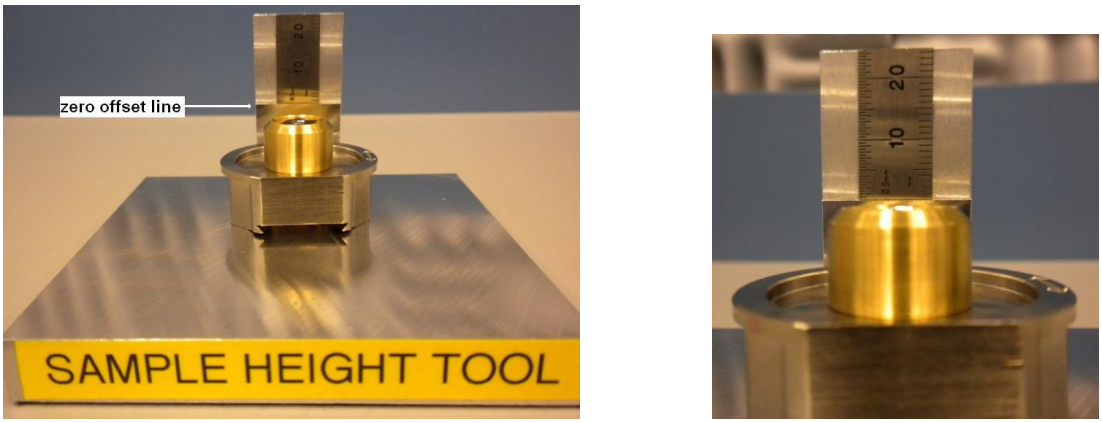

FIG. 2. Sample height tool. The right image shows a sample correctly flushed at the zero offset line.

II. Loading a specimen

1) Log on your session in the Facility Online Manager (FOM) software (icon on the desktop of the DATA computer). After a correct login, the SEM monitor should automatically turn on. If it does not, STOP YOUR WORK AND CONTACT A STAFF MEMBER. **Never modify any physical connections or push any system buttons. Doing so is unsafe for you and the instrument.**

2) Confirm that the stage is in the exchange position by checking that the EXCH POSN light on the airlock is **ON.** If not:
   a. Click the **OFF** button under **Observation** in the upper left section of the main window (see Fig. 3). The system diagram, located in the lower right corner of the main window, should show the beam stopped at the upper section of the SEM column. This means that the SEM column gate is closed, if not, please contact the tool instructor.
   b. Click the **Observation** button in the upper right section of the main window.
   c. Click the **Exchange Position** button in the SEM Monitor window.

FIG. 3. Main window of Graphical User Interface (GUI).

3) Before continuing with the next step, make sure that all the stage coordinates (X, Y, R and T) are "0.0", except for Z, which should be "38.0".

4) Turn on a "live" image of the chamber using the infrared (IR) camera. To do this, click on the "Windows" key in the SEM computer keyboard and select "IR Camera" icon.

5) Ordinarily, the airlock chamber is under high vacuum and the airlock chamber isolation valve is open. Before loading, the lights in the airlock buttons should be: VENT-off / EVAC-on / EXCH POSN-on / HLDR-off.

6) Press the VENT button for 2 seconds then release it. (The VENT light blinks; the isolation valve closes; N_2 gas vents into the airlock.)

7) When the VENT light stops blinking, unlock the airlock chamber by releasing the clasp. Open the airlock door.

8) **Lock the specimen holder into the clamp on the end of the exchange rod. (The specimen height above the top of the holder is limited to ~5 mm.) Make sure the flat side of the specimen holder lies perpendicular to the insertion direction (see figs. 4 and 5).**

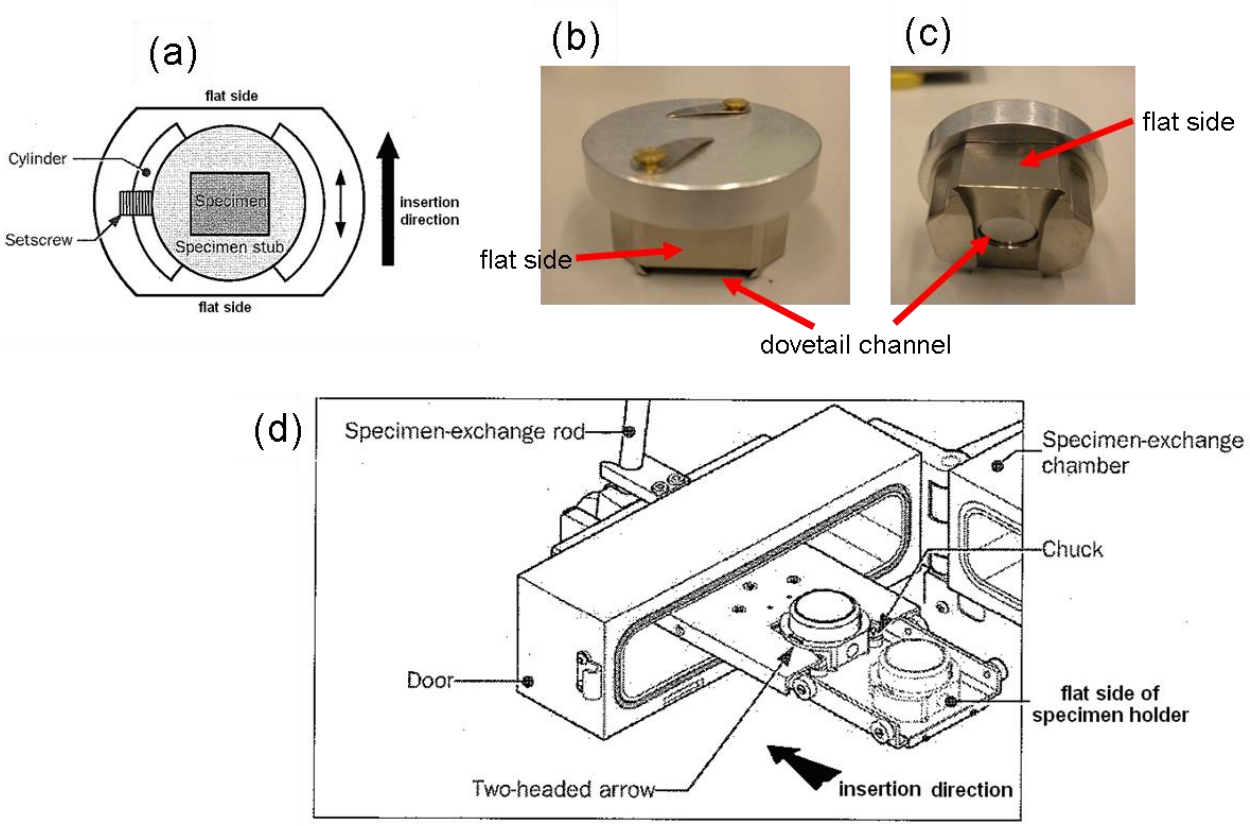

FIG. 4. Locking the sample holder in place in the airlock chamber. a) Top view of specimen holder. b) and c) Identification of "flat side" with respect to dovetail channel at the base of holder. The flat side is perpendicular to the length of the dovetail channel. d) Correct way of locking specimen holder with the holder's flat side perpendicular to the insertion direction. (Diagrams taken from JEOL's manual.)

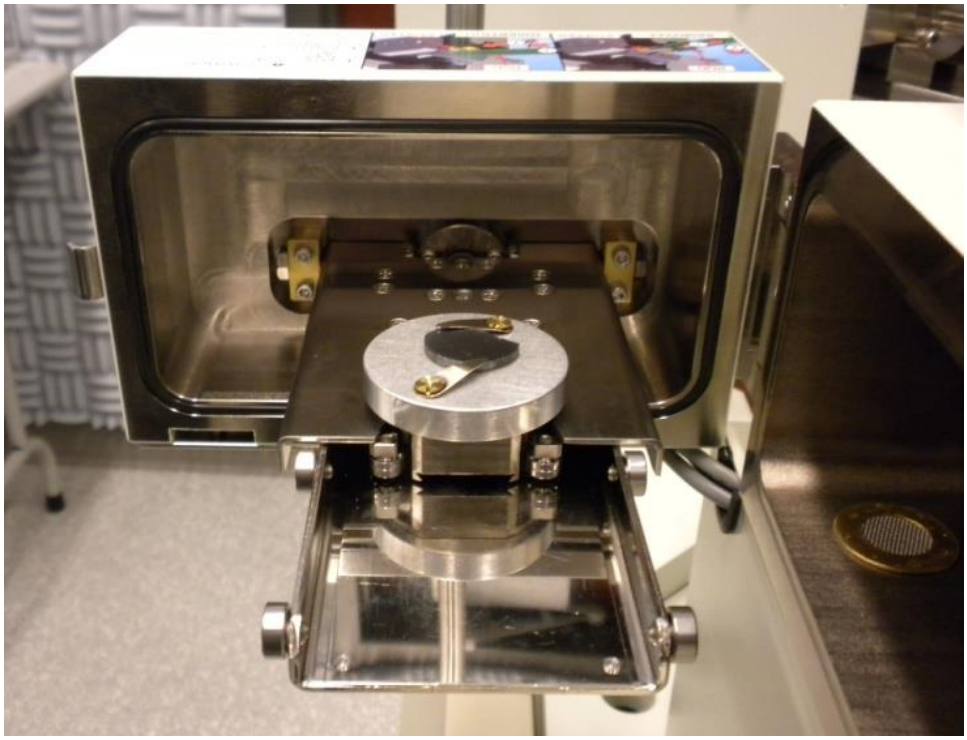

FIG. 5. Sample locked in place in the airlock chamber

IT IS VERY IMPORTANT TO CHECK THAT THERE ARE NO VERTICAL GAPS BETWEEN THE HOLDER AND THE CLIP MECHANISM. GENTLY PUSH DOWN ON THE HOLDER TO MAKE SURE IT SITS PROPERLY OVER THE CLIP.

9) Before closing the airlock chamber, check that the door's O-ring is free of dust and dirt and correctly positioned in the groove.

10) Close and lock the airlock chamber and press the EVAC button. (The EVAC light blinks; the airlock is pumped to high vacuum; the isolation valve opens.)

11) Wait until the EVAC light stops blinking. Now the lights in the airlock buttons should be: VENT-off / EVAC-on / EXCH POSN-on / HLDR-off. Also confirm that the system diagram (lower right of main window) indicates that the airlock chamber is under vacuum (gray color) and that the airlock isolation valve is open.

12) Lower the specimen-exchange rod horizontally **without pulling along its axis** (see Fig. 6). Once it is completely horizontal, the low pressure in the chamber may suck the rod in. This is normal, it will stop by itself due to friction.

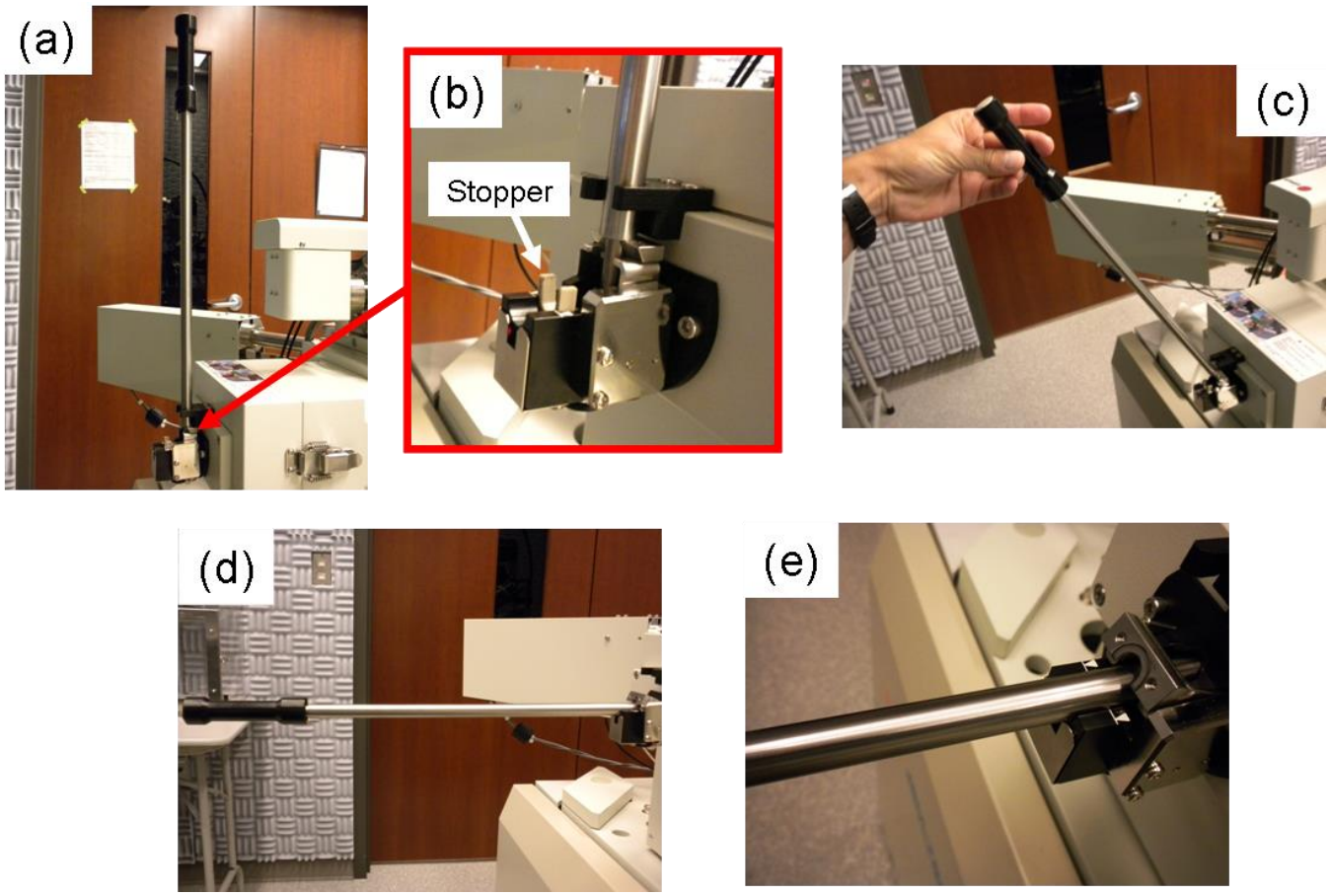

FIG. 6. Lowering of specimen-exchange rod. (a) Initial vertical position. (b) Detail of location of the plastic stopper. (c) Lowering of rod without pulling along its axis. (d) Fully horizontal position. Rod is held extended due to friction in sliding metal parts. Note that plastic stopper is pushed down when rod is horizontal as shown in (e).

13) Fully insert the specimen-exchange rod, keeping the holder horizontal, until you feel it come to a firm stop. Look at the chamber live image to detect this event. Then apply more force to lock the holder in the SEM stage (see Fig. 7).

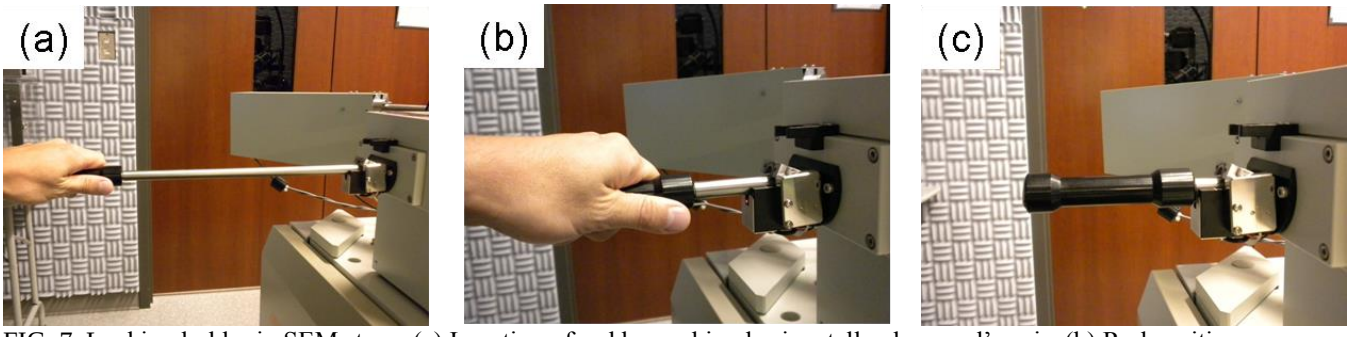

FIG. 7. Locking holder in SEM stage. (a) Insertion of rod by pushing horizontally along rod's axis. (b) Rod position when it first touches the SEM stage. (c) Rod position with holder locked in SEM stage.

14) After confirming that the HLDR light has lit up, **fully** retract the exchange rod horizontally until the plastic stopper snaps and comes up (see Fig. 8). Let the rod sit on the stopper and then tilt up the exchange rod **without pulling along its axis**. Now the lights in the airlock buttons should be: VENT-off / EVAC-on / EXCH POSN-on / HLDR-on.

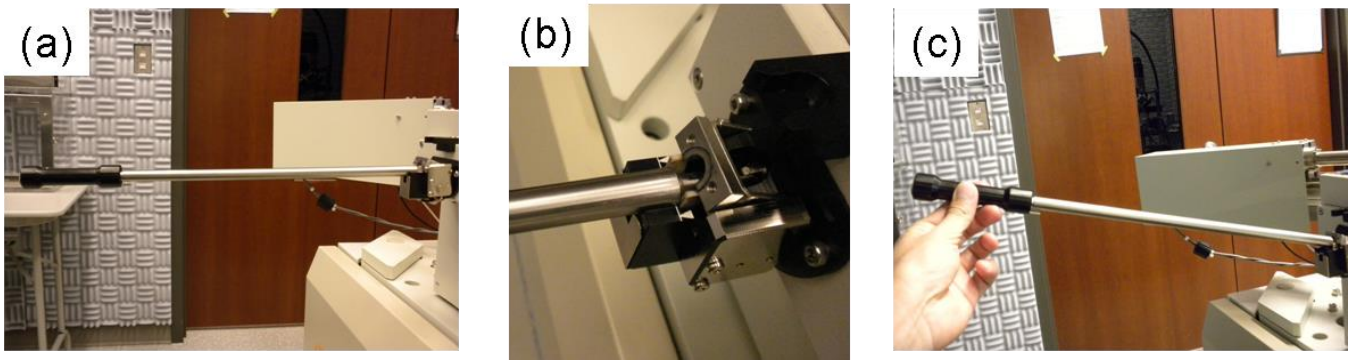

FIG. 8. Retracting exchange rod. (a) Fully retracted and resting on the plastic stopper. (b) Detail of rod resting on plastic stopper. C) Lifting of rod to its vertical position without using any force along rod's axis.

15) Click on the **Specimen Offset** button in the graphical user interface (GUI) and select from the list the holder you installed. If your sample has an offset (in mm) measured with the sample height tool (see Fig. 2), enter it in the *specimen surface offset* field of the specimen holder pop-up window (see Fig. 9).

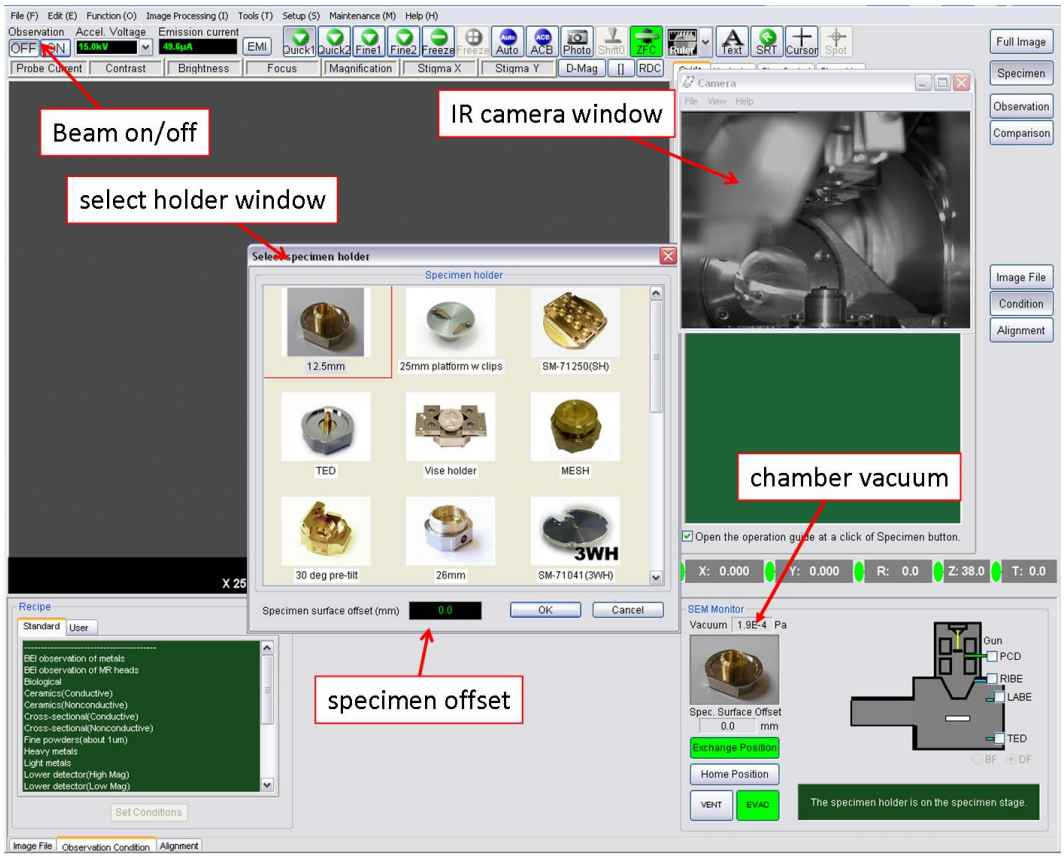

FIG. 9. Graphic user interface showing the specimen holder popup window.

III. Obtaining an image

1) Wait until the chamber vacuum is at $5x10^{-4}$ Pa or lower. Open the Gun Isolation Valve by clicking the ON button under Observation.

3) Select the SEI detector and SEM mode, and click on the working distance (WD) in the image info area of the GUI (see Fig. 10). **VERY IMPORTANT: Only click on WD when in SEM (high mag mode), never do it in LM (low mag mode).** Select 15 mm from the list and click "OK" in the window that pops up after clicking WD. This action will focus the beam to a WD of 15 mm and will bring the stage to Z=15.0+OFFSET. If the stage doesn't move, check that the ZFC button is "on" (green). Keep an eye on the movement in the IR camera window.

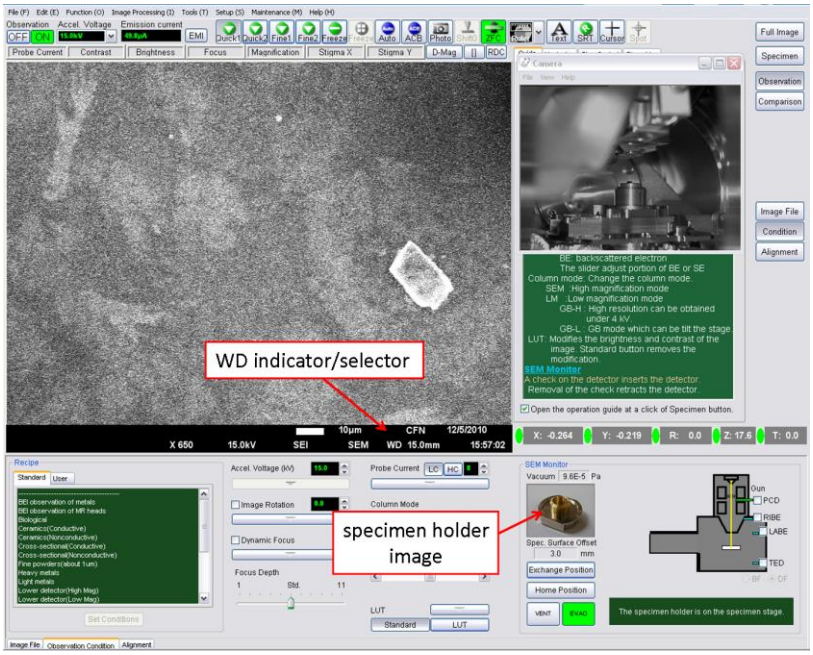

FIG. 10. Graphic user interface showing the position of the WD indicator/selector area.

4) Unfreeze the image, if necessary, by clicking on the FREEZE button in the knobset panel (see Fig. 11).

FIG. 11. Knobset panel, specimen stage control panel and trackball.

5) Find a feature in your sample by moving the stage using the trackball (see Fig. 11). If needed, select LM mode and, once you find the feature of interest, switch back to SEM. Adjust contrast and brightness using the autocontrast (ACB) button or the IMAGE CONTRAST and BRIGHTNESS knobs in the knobset panel (see Fig. 11). If the offset is correct, the image should be rather focused. Now, rotate the outer ring of the trackball until the image is in good focus. As you rotate the ring, the Z value changes. Make sure Z doesn't change by more than ±2 mm from its initial value. **If not sure about this step, please contact the tool instructor.**

6) Once the image is in focus, update the sample offset. For example, if your sample had an initial offset of 3 mm (Z=15.0+3.0=18.0 mm), and after focusing with the outer ring, Z=17.5 mm, then the new offset should be OFFSET=17.5 - 15.0 = 2.5 mm. Click on the sample holder image (see Fig. 9) and enter the new offset in the corresponding field.

7) Choose probe current setting. You may change the probe current by selecting the desired level in the probe current section of the GUI (right under the WD info area, see Fig. 10):
   a. For most secondary electron (SE) imaging choose low current (LC) mode with levels 1-10 (6-7 typical). The objective lens (OL) aperture should be set to #4.
   b. For analytical work, especially when using wavelength dispersive spectroscopy (WDS), choose high current (HC) mode with levels 11 – 20 and with the OL aperture set to #1.

   CAUTION.- Do not attempt to change the OL aperture without having been trained on this specific procedure by the tool instructor.
   CAUTION.- If you change the current setting, repeat steps 5 and 6 to update the sample offset.

8) Now, you are ready to navigate to the region of interest in your sample, and if necessary, change the beam parameters and the working distance**.**
   a. Use low magnification mode (LM) when necessary by pushing the LOW MAG button in the knobset panel. Navigate to the area of interest using the trackball.
   b. Use SEM mode in high magnification when possible. This is selected when the light of the LOW MAG button is off.
      CAUTION.- If you move the stage by more than 1 mm, repeat steps 5 and 6 to update the sample offset.
   c. Set WD to 4-6 mm for best resolution secondary electron (SE) imaging using the inlens detector (SEI), especially at low beam energies.
   d. Set WD≤8 mm for good resolution SE imaging using the low, in-the-chamber SE detector (LEI).
   e. Set WD=8 mm for EDS, and WD=15 mm for WDS work.

   CAUTION.- If you desire to change to a shorter WD, repeat steps 5 and 6 to update the sample offset.
   CAUTION.- Unless you have been authorized by the tool instructor, the minimum WD you can use is 4.5 mm. Note that EDS work is done at WD=8 mm and WDS work is done at WD=15 mm.
   NOTE.- The shortest WD for 30 keV and 15 keV is 6.5 mm and 4.5 mm, respectively. For beam energies ≤ 2 keV, the shortest WD can be 2 mm, however, make sure you have authorization from the tool instructor before setting WDs below 4.5 mm.

IV. Optimizing an image

1) Align the beam.
   a. Set magnification to ~10,000x - 50,000x using the MAGNIFICATION knob in the knobset panel.
   b. Focus the image (FOCUS knob in knobset panel) and correct astigmatism if necessary (see IV.2)
   c. Turn the wobbler on (WOBB button in knobset panel). If the image shifts, adjust X and Y knobs to stop image shifting
   d. Turn the wobbler off.

2) Astigmatism correction.
   a. Find a feature that has approximately circular shape using medium to high magnification.
   b. Using the FOCUS knob, check for astigmatism by going through over and under focus while looking for directionality of focus in the image (over and under focus directionality will be at right angles to each other).
   c. Stop focus at center of over and under focus (image may not be sharp but has no directionality of focus).
   d. Adjust the X and Y stigmation knobs (one at a time) and try to obtain an image as sharp as possible.
   e. Focus the image with the FOCUS knob and, if necessary, repeat steps b-e.
   f. If required, increase the magnification and repeat steps a-e.

V. Unloading a specimen

1) Click the **OFF** button under **Observation** in main window (see Fig. 12). The system diagram, located in the lower right corner of the main window, should show the beam stopped at the upper section of the SEM column.

2) Click the **Exchange Position** button in the SEM Monitor window.

FIG. 12. Main window of Graphical User Interface (GUI).

4) Before continuing with the next step, make sure that all the stage coordinates (X, Y, R and T) are "0.0", except for Z, which should be "38.0".

5) Turn on a "live" image of the chamber using the infrared (IR) camera. To do this, click on the "Windows" key in the SEM computer keyboard and select "IR Camera" icon.

6) Ordinarily, the airlock chamber is under high vacuum and the airlock chamber isolation valve is open. Before unloading, the lights in the airlock buttons should be: VENT-off / EVAC-on / EXCH POSN-on / HLDR-on.

7) Fully insert the specimen exchange rod until it "grabs" the specimen holder on the SEM stage. You can check this event in the IR camera image. These steps are describe in steps II.12 and II.13. Confirm that the HLDR light remains on

8) Fully retract the exchange rod as described in step II.14. Confirm that the HLDR light goes off.

9) Press the VENT button. (The VENT light blinks; the isolation valve closes; N_2 gas vents into the airlock.)

10) When the VENT light stops blinking, unlock the airlock chamber by releasing the clasp. Open the airlock door.

11) Remove the specimen. Close and lock the airlock chamber and press the EVAC button. (The EVAC light blinks; the airlock is pumped to high vacuum; the isolation valve opens.) The lights in the airlock buttons should be: VENT-off *I* EVAC-on *I* EXCH POSN-on *I* HLDR-off.

12) Close the IR camera window to increase the life of safe the life of the IR lamp.

13) Log off your session in the Facility Online Management (FOM) software. The SEM monitor should automatically turn off. If it does not, please contact the tool instructor.

V. Notes on using the TED detector

1) Make sure you have previously accurately determined the stage offset (see section III).
2) Go to a WD between 6-8 mm.
3) Verify that the vacuum level is in the mid 10^{-4} Pa range or better.
4) Click the **OFF** button under **Observation** in main window (see Fig. 12). This will isolate the vacuum in the e-beam column.
5) Insert the TED and wait until the vacuum level is back at $5x10^{-4}$ Pa or better. This will take a few minutes.
6) Once the vacuum level is appropriate, click the **ON** button under **Observation** in main window.

VI. Policy for mounting powder samples (including magnetic powder samples)

We need to be extra careful when mounting powder sample, especially magnetic.

1) Before mounting ANY powder sample, YOU NEED TO SHOW IT TO THE TOOL MANGER TO RECEIVE GREEN LIGHT TO GO ON. Once it is determined that it is safe for the system, you can repeat the mounting method as many times as you want on your own. In the next section you can find some tips on mounting powder samples, including magnetic powder samples.
2) The closest WD for magnetic powder samples is 8mm. You can't image these samples any closer.
3) Make sure you secure extremely tight any bulk magnetic sample to the sample holder to avoid any chance of having it fly onto the objective lens.

VII. Tips for mounting powder samples (including magnetic powder samples)

- A good general procedure is to cover an aluminum or carbon stub with carbon paint or silver paint/cement and quickly deposit a very small amount of powder on to the stub before the paint dries. Once dry, blow off any loose particles with compressed air. Remember that YOU CAN'T DO THIS IN 1L32, you need to do it in an approved lab here on in your own institution.

- Nano sized magnetic particles .- If the particles are nano sized and are relatively small in number, mounting them on a carbon stub by drying an alcohol suspension is OK. The weak force will keep them stuck to the stub. They can also be mounted on lacey or holey carbon TEM grids the same way (this implies using the TEM sample holder). Mounting on TEM grid reduces considerably the interaction volume allowing higher resolution for elemental mapping.

- Imaging large size (> 1 μm) magnetic particles.- This size particles cannot be mounted as in the previous bullet, because the objective lens (OL) flux will pull them onto the lens. For imaging these large particles, to study rough particle morphology for example, you need to

insure that they are FIRMLY stuck down in carbon tape with all of the loosely adhering particles blown off with compressed air or nitrogen. (You have to do this in your own lab, or an in approved lab in the CFN, not in 1L32!) For safety of the microscope, only use low mag (LM) mode for imaging these particles. In LM mode, the OL flux field is turned off.

- EDS of large size magnetic particles.- Mount these particles in a 1" or 1 ¼" inch standard epoxy mount (see for example, http://www.tedpella.com/material_html/mat1.htm). Polish the mount to expose surfaces of particles and then coat with carbon. This will give the best microanalysis conditions.

- VERY IMPORTANT! Mounting samples of the kind described in this section CANNOT be done in lab 1L32. Ask the tool manager to give you a holder for you to mount these samples in an appropriate lab in the CFN or in your own institution.